ACTIVE OXIDATION OF A UHTC-BASED CMC

David E. Glass and Scott C. Splinter
MS 190, NASA Langley Research Center, Hampton, VA 23681

ABSTRACT

The active oxidation of ceramic matrix composites (CMC) is a severe problem that must be avoided for multi-use hypersonic vehicles. Much work has been performed studying the active oxidation of silicon-based CMCs such as C/SiC and SiC-coated carbon/carbon (C/C). Ultra high temperature ceramics (UTHC) have been proposed as a possible material solution for high-temperature applications on hypersonic vehicles. However, little work has been performed studying the active oxidation of UHTCs. The intent of this paper is to present test data indicating an active oxidation process for a UHTC-based CMC similar to the active oxidation observed with Si-based CMCs. A UHTC-based CMC was tested in the HyMETS arc-jet facility (or plasma wind tunnel, PWT) at NASA Langley Research Center, Hampton, VA. The coupon was tested at a nominal surface temperature of 3000°F (1650°C), with a stagnation pressure of 0.026 atm. A sudden and large increase in surface temperature was noticed with negligible increase in the heat flux, indicative of the onset of active oxidation. It is shown that the surface conditions, both temperature and pressure, fall within the region for a passive to active transition (PAT) of the oxidation.

INTRODUCTION

Hypersonic and re-entry vehicles experience extremely high temperatures due to the aerodynamic heating of flight. In many cases, these temperatures can exceed the 3000°F (1650°C) range where Si-based composite materials such as coated carbon/carbon (C/C) and C/SiC are acceptable for extended use. For these higher temperatures, far exceeding the 3000°F (1650°C) range, ultra high temperature ceramics (UHTC) and UHTC-based composites have been proposed. Many of the UHTCs and UTHC-based composites contain Si, which can accelerate the transition to active oxidation and the resulting increased erosion. The active oxidation of Si-based composites is well characterized, but the active oxidation of UHTCs and UHTC-based composites has not been well characterized. A large temperature increase characteristic of the passive-to-active transition has been observed in an arc-jet (or PWT) test of a UHTC at NASA Ames [1]. It is suspected that the large temperature increase is due to the transition to active oxidation, but cannot be confirmed due to uncertainty of the test conditions.

In this effort, a UHTC-based composite provided by Starfire Systems was tested in the HyMETS arc-jet facility (or PWT) at NASA Langley Research Center in Hampton, VA. Tests were performed on multiple coupons. In one of the tests, as the facility conditions were held relatively constant, a sudden and sharp increase in the surface temperature was observed, indicating the transition to active oxidation.

HYMETS FACILITY DESCRIPTION

The HyMETS facility was installed at NASA Langley Research Center in 1968 as a 100 kW segmented-constrictor-arc-heated wind tunnel [2] and throughout the 1970's, 80's, 90's, and early 2000's, was used primarily for emissivity, catalycity, and dynamic oxidation testing of metals and coatings for hypersonic vehicles [3-8]. The range of test conditions for the HyMETS facility during this time is presented in Table 1. In 2005, the facility was taken offline and the old 100 kW power supply, which only functioned at 50 kW at that time, was upgraded to a 400 kW power supply with programmable-logic-control (PLC) [9]. Since then, the facility has been used as part of the Return To Flight Reinforced Carbon/Carbon On-Orbit Repair Project to characterize the Non-oxide Adhesive Material (NOAX) concept for crack and coating repair [10]. The facility has also

been used to prove the concept of receiving an optical signal through heat-shield materials in flight-like conditions before applying the new technique in more complicated and costly arc-jet facilities. The HyMETS facility is used primarily for the characterization, cycling, and screening of candidate hypersonic materials, but it can also be used for performing research and development efforts on hypersonic plasma flow diagnostics.

Table 1: HyMETS Historic Range of Test Conditions

Coupon surface temperature, °F (°C)	1472-2732 (800-1500)
Coupon stagnation pressure, atm	0.004-0.008
Free stream Mach number	3.5
Free stream enthalpy, Btu/lbm (J/g)	1719-4730 (4000-11,000)
Cold wall heat flux, Btu/ft^2-sec (W/cm^2)	70-400 (80-454)

CONFIGURATION

The HyMETS facility, shown in Figure 1, is configured with a segmented-constrictor-dc-electric-arc-heater as an arc-plasma generator. The arc-plasma generator is mounted on the outside of the test chamber door and consists of a copper cathode with tungsten button emitter, 32 electrically-isolated copper segment constrictors with a 0.5-in-diameter (1.3 cm) bore, a sterling-silver divergent-ring anode, and a copper convergent-divergent Mach 5 nozzle with a 0.5-in-diameter (1.3 cm) throat, 2.5-in-diameter (6.35 cm) exit plane, and a half-angle of 8°. All arc-plasma generator components are water cooled via jackets or internal passageways.

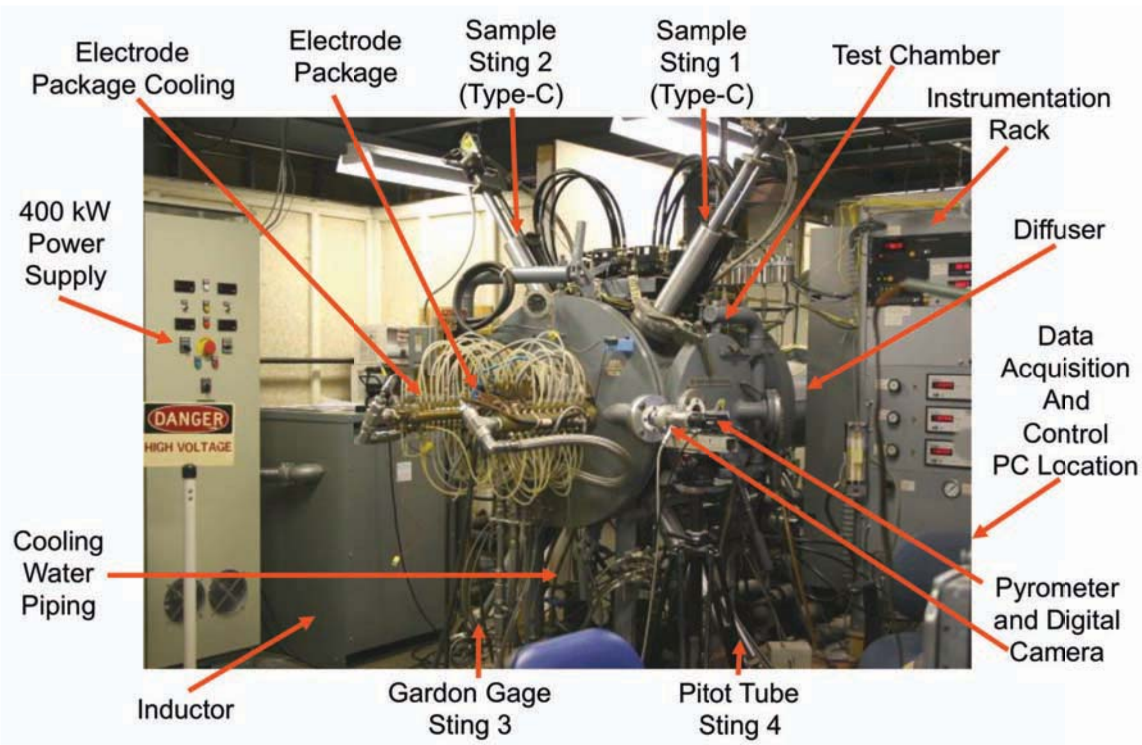

Figure 1. Photograph of HyMETS arc-jet (or PWT) facility.

Test gasses are injected tangentially into the bore of the arc plasma generator at 6 discrete locations, and are heated by a high-voltage arc column maintained between the electrodes to create the high-temperature ionized arc-plasma flow. The electric arc is spin-stabilized in the arc-plasma generator by the vortex motion of the injected test gasses. The test gases used in the arc-plasma generator are supplied by several compressed gas cylinders, and can be custom

mixed to any desired atmosphere. Adjustable volume percentages of N_2 and Ar are used as shield gases near the cathode and anode, respectively, to protect the electrodes from rapid oxidation. The compressed gas cylinders can provide a run time of 1 hr at the high mass flow rates, or several hours at lower mass flow rates.

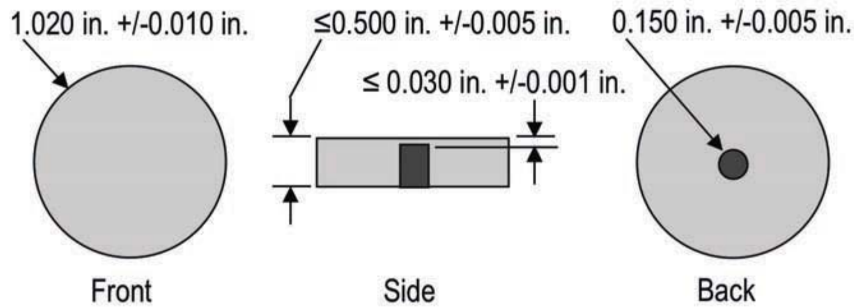

Figure 2. Schematic drawing of the test coupon.

Power to the electrodes is provided by a 400 kW power supply with programmable-logic-control (PLC) and induction filter. The arc-plasma flow from the arc-plasma generator is accelerated through the nozzle and exhausted into a 2-ft diameter (0.61 m), 3-ft-long (0.91 m) vacuum test chamber where it stagnates on a 1-in. (2.54 cm) diameter by 0.5-in-thick (1.3 cm) test coupon and is then captured by a collector nozzle with an 8-in-diameter (20 cm) inlet plane, a 6-in-diameter (15 cm) constant cross-section diffuser, and a coiled-copper tubing heat exchanger which are coupled to a two-stage, continuous-flow, high-mass-capacity, mechanical pumping system to decelerate, cool, and evacuate the arc plasma flow from the test chamber. The collector, diffuser, heat exchanger, and mechanical pumping system provide for proper expansion of the arc-plasma flow from the nozzle. The entire facility is cooled by a 150-ton re-circulating chiller with associated booster pumps and heat exchangers.

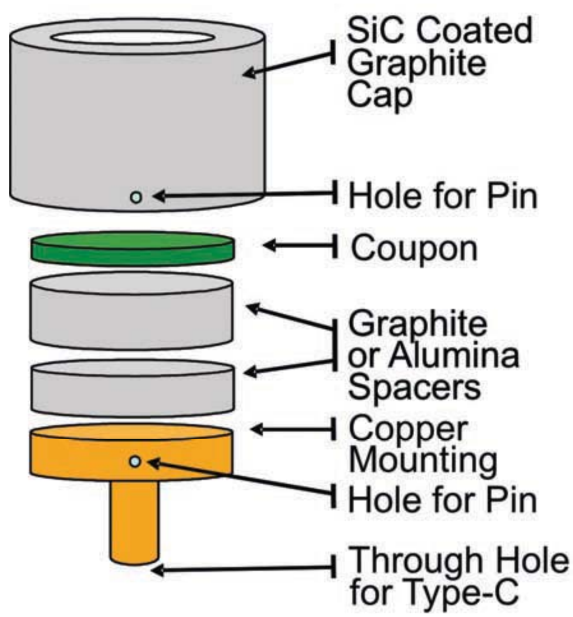

Figure 3. Schematic drawing of test coupon and holder components.

The test coupon is 1-in. (2.54 cm) diameter and is required to be less than 0.5-in. (1.3 cm) thick. A schematic of the test coupon is shown in Figure 2. The test coupon is held in a SiC coated graphite holder by a copper mounting holder. Spacers are placed between the test coupon and the copper mounting holder. A pin is placed through the graphite cap and the copper mounting holder to hold all the parts in place. The copper holder has a hole (axial direction) in it through which a thermocouple can be placed. The thermocouple can either be embedded in the

test coupon or in contact with the back surface. A schematic drawing of the different components of the holder assembly is shown in Figure 3, and a photograph of each of the components is shown in Figure 4. The test coupon is the second from left in Figure 4.

Figure 4. Photograph of test coupon and holder components.

The HyMETS facility is operated by LabVIEW [11] software from a PC workstation and a touch-screen operations module. Test conditions are controlled by adjusting the current and test gas mass flow rate setpoints within LabVIEW for the arc-plasma generator, and can be operated either manually or automatically allowing for the generation of test profiles.

INSTRUMENTATION

Test coupons and instrumentation probes are mounted on four water-cooled injection stings arranged symmetrically around the inside circumference of the test chamber a distance of 1.5 in. (3.8 cm) from the nozzle exit, and are inserted into the arc plasma flow by the touch-screen operations module. The instrumentation used in the HyMETS facility consists of a Pitot tube which measures stagnation pressure, a SiC probe [12] which measures semi-catalytic hot wall heat flux, a Gardon Gauge [13] and a Copper Slug Calorimeter [14] which measure fully-catalytic cold wall heat flux, and a Teflon® Slug Calorimeter [14-15] which measures non-catalytic cold wall heat flux. The two lower injection stings house the Gardon Gauge and the Pitot tube shown in Figure 5. The two upper injection stings house Type-C thermocouples and are used to insert test or calibration coupons into the arc plasma flow (shown in Figure 6), and can also be used to house the Copper and Teflon® Slug calorimeters and/or the SiC probe.

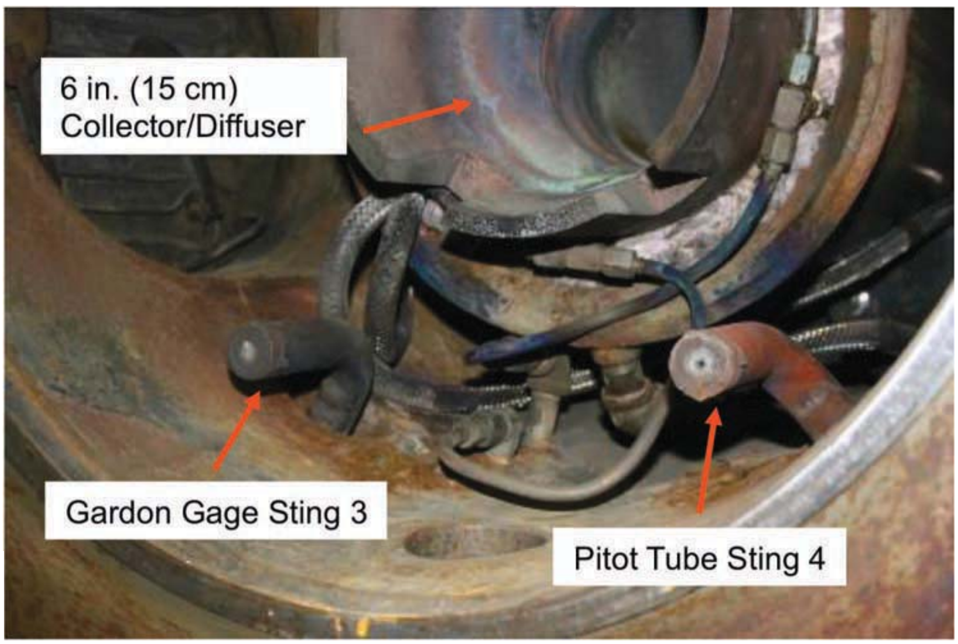

Figure 5. Photograph of Gardon type heat flux gage and Pitot tube.

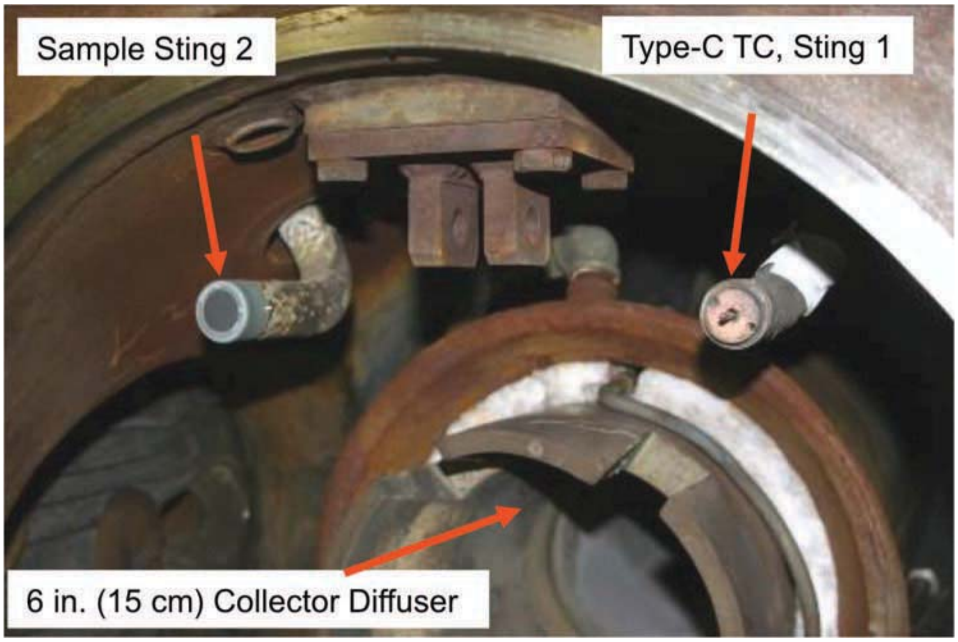

Figure 6. Photograph of coupon and thermocouple on sting 1 and 2.

A two-color (ratio) pyrometer and full color digital video camera (shown in Figure 7) with variable exposure settings was positioned outside the test chamber and remotely aimed through a viewport on the test chamber door during testing. The bulk enthalpy of the plasma flow was determined using a thermopile to measure the differential temperature across the inlet and outlet of the cooling water manifolds for the arc-plasma generator, and flow meters to measure the mass flow rates for both the cooling water and the arc plasma generator test gas [12]. The stagnation pressure, various heat fluxes, bulk enthalpy, current, voltage, and mass flow rate data are recorded at 100 Hz.

Figure 7. Photograph of digital camera and 2-color pyrometer used in HyMETS.

RESULTS AND DISCUSSION

Starfire Systems, Malta, NY, provided UHTC-based coupons with a pre-ceramic polymer based coating. The test coupons were 1-in-diameter and ~0.16-in. thick. The mass flowrate in the arc jet (or PWT) was set to achieve a pressure of 0.026 atm (19.26 Torr) at a temperature of 3000°F (1650°C) or 3200°F (1760°C), which were the desired conditions. Coupons were to be tested up to 10 cycles, with each cycle lasting 20 min.

CALIBRATION

Four of the coupons (SF-CMC-1.0D-07 through SF-CMC-1.0D-10) were used to help determine the desired arc-jet test conditions. The first run with a Starfire coupon attempted to determine the arc conditions to achieve the 3000°F (1650°C) surface temperature and 0.026 atm stagnation pressure. The second run was used to correct the pyrometer (based on coupon surface properties) by correlating it to a thermocouple inserted through the back surface. The thermocouple was Type-C and was approximately 0.030 in. (0.76 mm) from the heated surface, and was assumed to read the approximate surface temperature. The actual thickness of the material between the thermocouple and the outer surface was not measured. No corrections were made to the thermocouple readings. A third run was used to re-establish the arc conditions, and a fourth run was used to generate preliminary data on cycles at the established set points.

CYCLIC TESTING

Four coupons were then tested in the HyMETS facility. The first three coupons tested, SF-CMC-1.0D-01, SF-CMC-1.0D-02, SF-CMC-1.0D-03, each experienced significant weight loss prior to the goal of 1, 5, and 10 cycles, respectively, as seen in Table 2. Coupon -01 (abbreviated notation for SF-CMC-1.0D-01) had a thermocouple inserted from the back surface, while coupons -02 and -03 had thermocouples in contact with the back surface. The temperatures in Table 2 are front surface temperatures from a pyrometer that had been corrected via a thermocouple in prior tests. Testing on coupon -02 was stopped after the third (of planned five) test due to the significant weight loss. On coupon -03, the top surface delaminated during preparation for the second test, halting further testing on that coupon.

An attempt was then made to avoid the suspected active oxidation by decreasing the temperature and/or increasing the pressure. Coupon -04 survived ten 20 min cycles, with each cycle reaching a nominal surface temperature of 3040°F (1670°C) and a nominal pressure of 0.04 atm. The weight loss of coupon -04 is shown in Table 2. Cycle 4 was aborted prior to 20 min. due to low N_2 pressure supply for the HyMETS facility, resulting in reduced mass flow and pressure in the arc jet. The temperatures were obtained from a pyrometer that had been corrected via a thermocouple.

Table 2: Starfire Systems Test Results

Specimen ID	Cycle	Temp. °F (°C)	Pressure, atm (Torr)	Cycle Time, min.	Initial Weight, g	Final Weight, g	% Weight Loss	% Total Weight Loss
SF-CMC-1.0D-01	1	~3000 (~1649)	~0.0258 (~19.64)	20	6.6219	5.8789	11.2	11.2
SF-CMC-1.0D-02	1	~3200 (~1760)	~0.0247 (~18.80)	20	6.2485	4.7845	23.4	23.4
	2	~3230 (~1776)	~0.0267 (~20.29)	20	4.7845	4.1717	12.8	33.2
	3	~3250 (~1787)	~0.0268 (~20.33)	20	4.1717	3.3967	18.6	45.6
SF-CMC-1.0D-03	1	~3225 (~1773)	~0.0264 (20.04)	20	6.0908	4.6955	22.9	22.9
SF-CMC-1.0D-04	1	~3000 (~1649)	~0.0373 (~28.37)	20	5.8392	5.5049	5.7	5.7
	2	~3050 (~1676)	~0.0406 (~30.87)	20	5.5073	5.3327	3.2	8.7
	3	~3050 (~1676)	~0.0398 (~30.22)	20	5.3327	5.1812	2.8	11.3
	4	~3060 (~1682)	~0.0387 (~29.42)	18.67	5.1812	5.0501	2.5	13.5
	5	~3040 (~1671)	~0.0402 (~30.54)	20	5.0530	4.8806	3.4	16.4
	6	~3030 (~1665)	~0.0387 (~29.39)	20	4.8777	4.6005	5.7	21.2
	7	~3040 (~1671)	~0.0410 (~31.13)	20	4.6005	4.4156	4.0	24.4
	8	~3030 (~1665)	~0.0404 (~30.73)	20	4.4177	4.2251	4.4	27.6
	9	~3060 (~1682)	~0.0416 (~31.65)	20	4.2251	4.0070	5.2	31.4
	10	~3145 (~1729)	~0.0408 (~31.00)	20	4.0070	3.5553	11.3	39.1

The front surface of coupon -04 is shown in Figure 8 prior to the first test, and after 1, 5, and 10 cycles. The photographs were taken while the coupons were mounted in the sting. The front surface temperature during cycle 1 was approximately 3000°F (1650°C) as read by the pyrometer. The back surface temperature, read by a spring-loaded thermocouple on the back surface, was ~2500°F (1370°C). The thickness of the coupon was ~0.16 in. (0.41 cm).

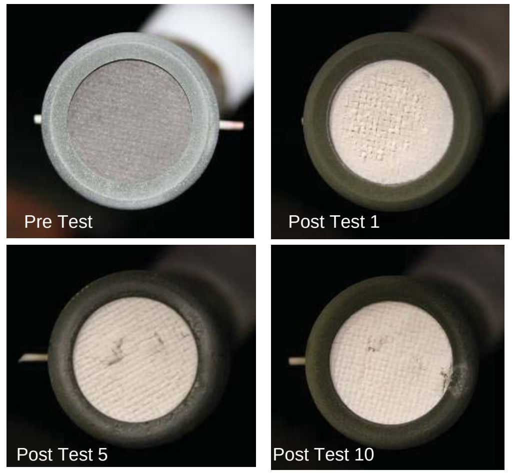

Figure 8. Photographs of front surface of -04 pre test, and after cycles 1, 5, and 10.

Starting with cycle 2, the surface temperature experienced a slight decrease after reaching a maximum value. This effect can be seen in Figure 9, which shows the front surface pyrometer reading. The "bump" in surface temperature lasted ~150 sec. The duration of the "bump" in surface temperature increased with each cycle, and by cycle 9, the "bump" lasted ~550 sec, as shown in Figure 10. Cycles 1 and 10 did not experience the "bump" in surface temperature. The cause of the bump and its increasing duration is unknown. In addition to the pyrometer reading of the front surface temperature, the back surface temperature was read via a spring loaded-thermocouple, and is shown in Figure 9. The current and voltage are also indicated on Figure 9, and are kept relatively uniform after the initial ramp up. Air, nitrogen (N2), and oxygen (O2) flow rates and set points are also included on the figure.

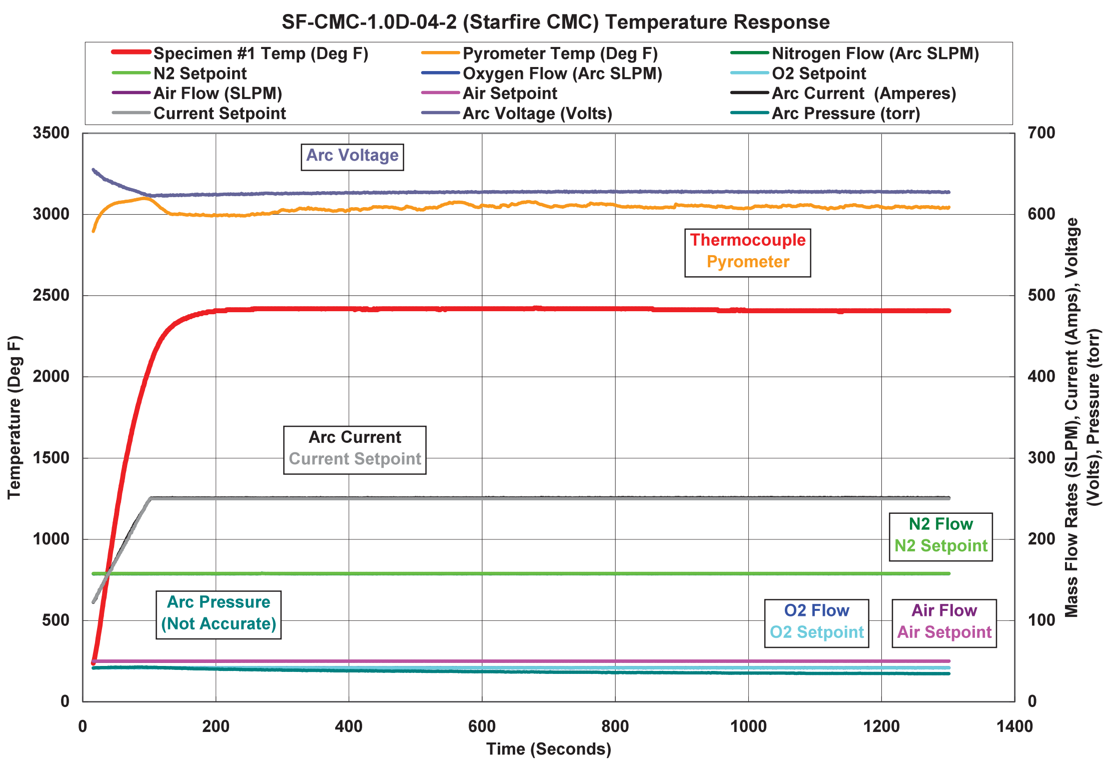

Figure 9. Test conditions for test coupon -04, cycle 2.

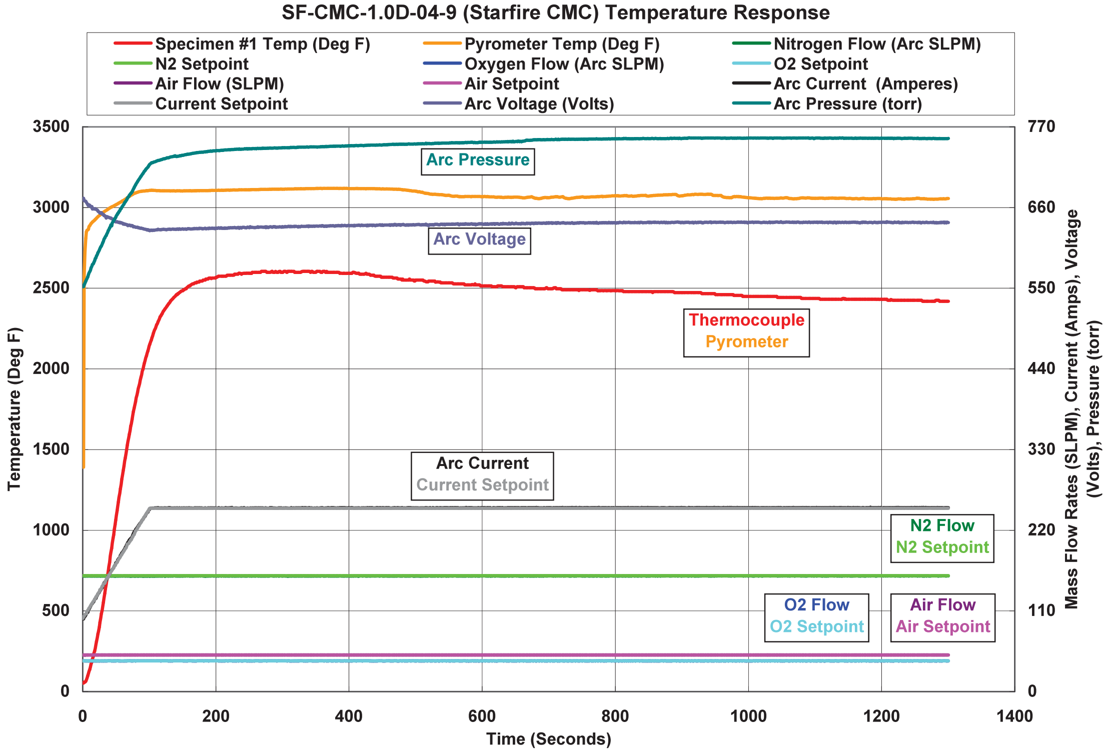

Figure 10. Test conditions for coupon -04, cycle 9.

At the end of cycle 5, a small portion of the back surface delaminated. After the next cycle, the entire back surface delaminated. Another large section of the back surface delaminated after cycle 10. A photograph of the back surface after cycle 10 is shown in Figure 11. It appears that the back of the coupon adhered to the alumina spacer. The delamination at the back surface contributed to the higher weight loss during this 10th and final cycle.

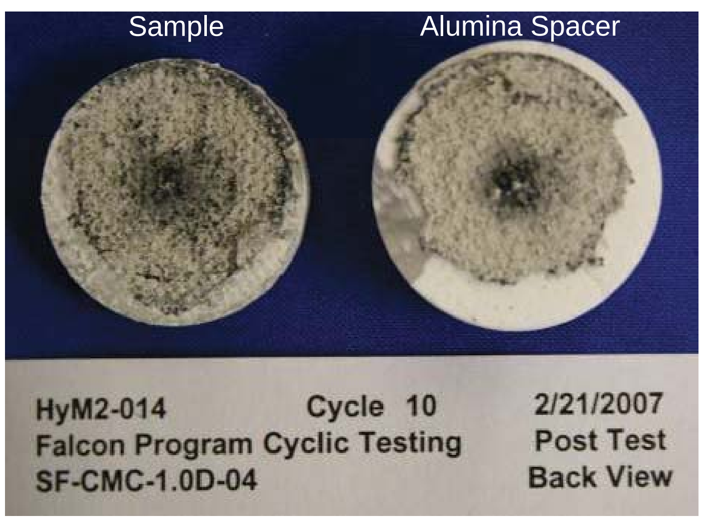

Figure 11. Photograph of the back surface of coupon -04 after cycle 10.

After Starfire coupon -04 was tested, the HyMETS facility was shut down for upgrades. After the facility was started back up, Starfire coupon -05 was tested in a similar manner to compare the facility pre- and post- upgrades. Prior to facility shut down, 250 Amps, 620 V, and 155.5 kW were required to reach surface temperatures of ~3000°F (1650°C) on the Starfire material. After the facility was started again, 300 Amps, 556 V, and 166.8 kW were required to reach

temperatures of ~3000°F (1650°C). The temperature, pressure, and weight loss from the Starfire -05 coupon runs are shown in Table 3.

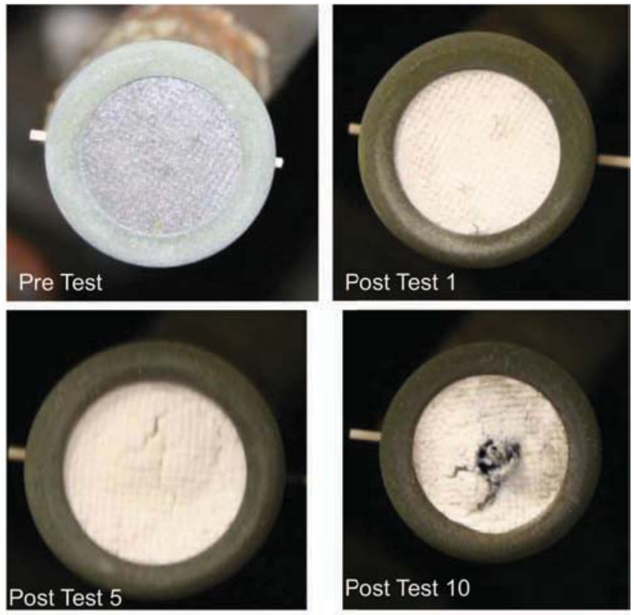

Figure 12. Photographs of front surface of Starfire -05 pre test, and after cycles 1, 5, and 10.

Starfire coupon -05 was tested for ten 20-minute cycles. Photographs pre-test, and after 1, 5, and 10 cycles are shown in Figure 12. During cycle 1, the heat flux started out at 172 Btu/ft^2-sec (195 W/cm^2), and was ramped up to 600 Btu/ft^2-sec (681 W/cm^2), as indicated by the current and voltage in Figure 13. The temperature experienced a much stronger oscillation than seen previously, and the coupon lost over 11% of its weight during the first cycle. Again, the pyrometer recorded the heated surface temperature, and the thermocouple recorded the back surface temperature. Argon gas was added to the flow (8%). On subsequent cycles, the final (steady state) heat flux was less than the 600 Btu/ft^2-sec (681 W/cm^2) on cycle 1, varying between 350 – 540 Btu/ft^2-sec (397 – 613 W/cm^2). The arc current and voltage (and thus heat flux) were set such that the pyrometer reading of the outer surface temperature was ~3000°F (1650°C).

Table 3: Run Data for SF-CMC-1.0D-05

Cycle	Temp. °F (°C)	Pressure, atm (Torr)	Heat Flux, Btu/ft^2-sec	Cycle Time, min.	Initial Weight, g	Final Weight, g	% Weight Loss	% Total Weight Loss
1	~3000 (~1649)	~0.043 (~32.28)	600	20	5.8259	5.1421	11.7	11.7
2	~3050 (~1677)	~0.04 (~30.13)	541	20	5.1421	4.8855	5.0	16.1
3	~3050 (~1677)	~0.04211(~32)	351	20	4.8855	4.3725	10.5	24.9
4	~3060 (~1682)	~0.03776 (~28.7)	521	20	4.3694	4.0214	8.0	31.0
5	~3050 (~1677)	~0.03817 (~29.01)	504	20	4.0214	3.7012	8.0	36.5
6	~3050 (~1677)	~0.04132 (~31.4)	530	20	3.6903	3.4107	7.6	41.5
7	~3090 (~1698)	~0.03454 (~26.25)	438	20	3.4107	3.2085	5.9	44.9
8	~3100 (~1704)	~0.04121 (~31.32)	509	20	3.2085	3.0473	5.0	47.7
9	~3100 (~1704)	~0.0407 (~30.93)	500	20	3.0441	2.8668	5.8	50.8
10	~3100 (~1704)	~0.0288 (~21.89)	503	20	2.8668	2.7555	3.9	52.7

The heat flux was measured via a Gardon Gage. However, the Gardon Gage had not yet been calibrated against a slug calorimeter. The heat flux values were thus qualitative in nature, and indicate the repeatability of the test conditions and the change in magnitude of the heat flux with different diffusers and collectors. Though the coupon holders were not water cooled, the sting was water cooled. It is uncertain how much heat was lost through the cooled sting. In summary, the heat flux values should be taken as qualitative, and showing trends, not as absolute numbers.

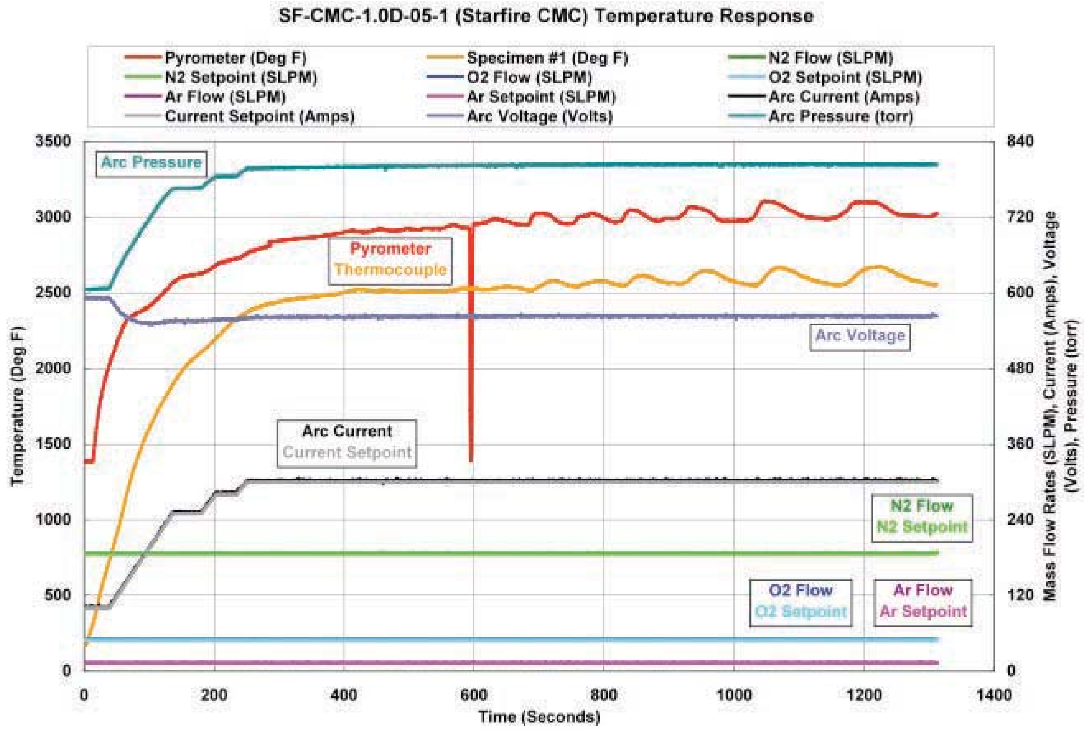

Figure 13. Temperature response for cycle 1 of SF-CMC-1.0D-05.

A plot of weight loss versus time for coupon -05 is shown in Figure 14. As shown in Table 3, the cumulative weight loss is over 50%. The rate of weight loss is relatively smooth over the 200 minutes of exposure.

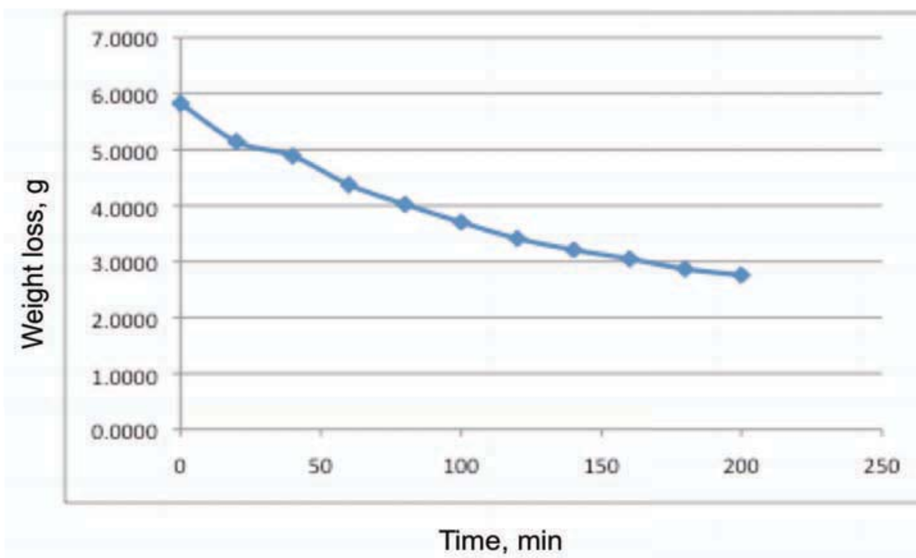

Figure 14. Plot of weight (g) versus time (min) for the SF-CMC-1.0-05 coupon.

ACTIVE OXIDATION

A rapid increase in surface temperature was observed during testing of several of the Starfire coupons. It is suspected that the coatings entered an active oxidation regime [16-17]. Figure 15

shows the data from the test of -08. The intent of this test was to determine the arc-jet parameters to achieve a surface temperature of ~3000°F (1650°C). A thermocouple was inserted in a hole drilled through the back surface of the coupon, and was estimated to be 0.03 in. (0.76 mm) from the heated surface. As can be seen in Figure 15, the thermocouple indicates a rapid increase in temperature while the voltage and current remained relatively uniform. The jump in temperature, indicated by the red circle on the figure, corresponds to a temperature of ~3300°F (1815°C) and a stagnation pressure of ~0.0015 atm, corresponding to an oxygen partial pressure of ~0.00031 atm. The sudden and large temperature increase has been noticed previously at different arc-jet (PWT) facilities. [16]

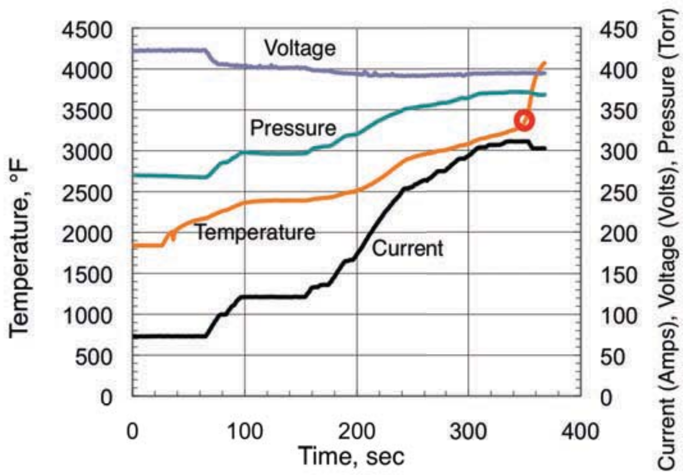

Figure 15. Arc-jet data showing rapid increase in surface temperature of SF-CMC-1.0D-08.

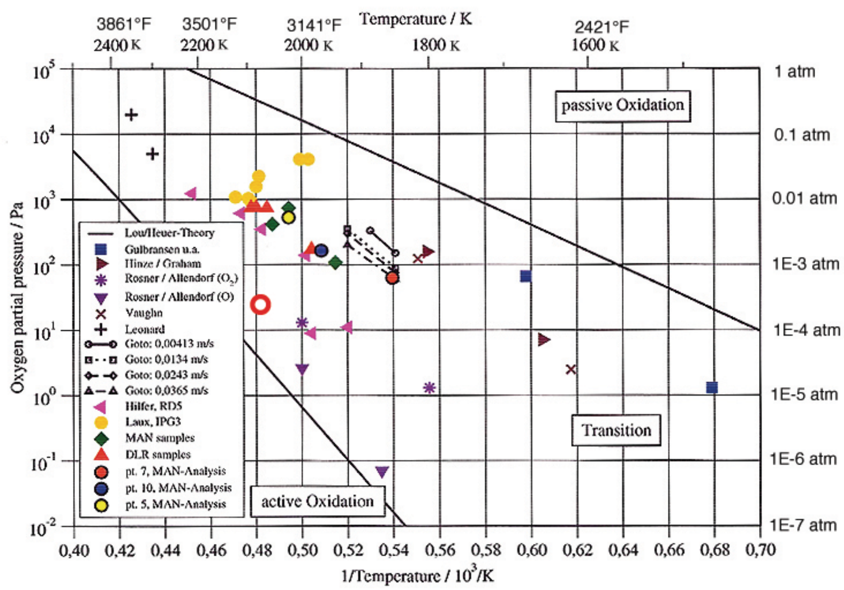

Figure 16. Transition from active to passive oxidation [18].[1]

Figure 16 is a compilation of data showing the transition from passive to active oxidation. The data point indicated by the red circle in Figure 15 is shown on Figure 16. It can be seen that the temperature and pressure for the HyMETS Starfire test lie in the transition region, implying that the jump in temperature corresponds to active oxidation.

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Results of an arc-jet (PWT) test of MT Aerospace C/SiC material performed in the German PWK2 facility are shown in Figure 17. As shown in the HyMETS data (Figure 15), the current to the facility remained relatively constant, and the surface experienced a sudden increase in temperature.

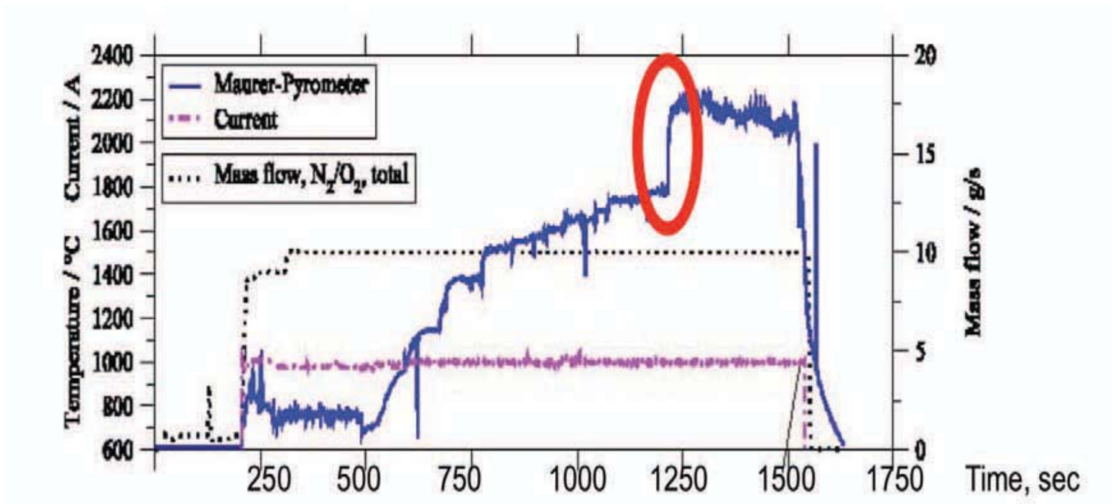

Figure 17. Arc-jet testing of MT Aerospace C/SiC in the German PWK2 facility [18].[2]

CONCLUDING REMARKS

Arc-jet (or PWT) testing of a UHTC-based composite in the NASA Langley HyMETS facility demonstrated the sudden and large surface temperature increase that has been observed with Si-based composites such as C/SiC. The temperature increase occurred with the power to the arc jet remaining relatively constant. The jump in surface temperature with a relatively uniform heat flux is indicative of the transition from passive to active oxidation of the UHTC-based composite material.

ACKNOWLEDGMENTS

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